High Spatial Resolution Infrared Microscopy with a Solid Immersion Lens

Infrared (IR) microscopy is a widely used, powerful technique for the characterization of spatial variations in chemical composition for complex, heterogeneous materials. This technique couples the high degree of chemical specificity that underlies the utility of macroscopic IR spectroscopy with the microscale spatial resolution possible with modern IR optics. Sample analyses of this type are particularly useful in establishing correlations between macroscopic performance properties (for example mechanical and chemical stability) and material microstructure, and thus play a useful role in the rational design of high-performance materials. One drawback of IR microscopy is that the attainable spatial resolution is relatively poor compared to that of conventional microscopy at visible wavelengths due in part to longer IR wavelengths and in part to sources and optics that are inferior to their visible counterparts. This limits the class of problems that are amenable to analysis with IR microscopy.

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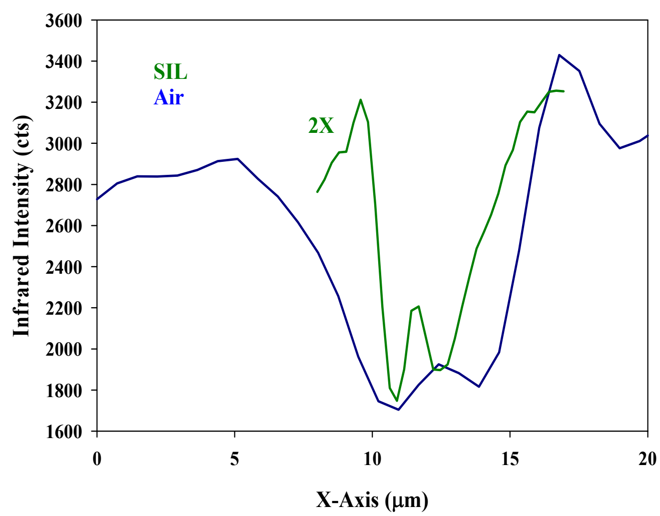
As technologically relevant materials increasingly have sub-micrometer critical dimensions, the demand for techniques that improve the attainable spatial resolution of IR microscopy will continue to grow. Solid immersion lens (SIL) imaging involves the use of a lens (usually hemispherical) made from a material with a high index of refraction, n. Converging light impinging on the curved surface of the hemisphere forms an aberration-free focus at the center of the bottom surface of the hemisphere. The diffraction limit for imaging resolution is then decreased by a factor of n over the limit in air. This improvement can be quite significant, particularly in the IR, where commonly used materials, such as zinc selenide (ZnSe) and germanium (Ge), have refractive indices in the range 2 to 4. Because the focus forms at the surface of the lens, the specimen is placed either in contact or close proximity (≈100 nm) to the lens. This technique has been widely explored in the visible spectral region for optical data storage applications but little effort has been directed toward its use for IR chemical imaging.

A custom, imaging IR microscope based on a ZnSe SIL, a broadband IR laser source, and a focal plane array detector has been constructed at NIST and its initial imaging performance evaluated.

The microscope was first characterized for imaging in air with standard resolution test targets and test samples consisting of 2.5 µm diameter polystyrene (PS) beads dispersed on a gold film. The demonstrated resolution was near the diffraction limit for the 3.4 µm imaging wavelength utilized. The resolution targets were imaged through the ZnSe SIL and the expected increase in system magnification (n) was confirmed. The PS beads were also imaged through the SIL, where the SIL was placed in contact with the specimen. The resolution enhancement was evaluated by comparison of the apparent sizes of the beads in the air and SIL images. Cross-sections of a 2.5 µm diameter PS bead imaged at λ=3.4 µm in air (blue trace) and through a ZnSe SIL (green trace) are shown in the graph below. Comparison of the widths of these features following a standard deconvolution procedure reveals a resolution increase factor of 2.5, in good agreement with the expected value of 2.4 (n_{ZnSe} at 3.4 µm). These results suggest that incorporation of a SIL into an IR microscope is a promising approach to increasing the attainable spatial resolution of IR microscopy and extending its utility to smaller length scales.



Impact: Planning is underway for the evaluation of the IR SIL microscope with microstructured polymer samples provided by a collaborator at Dow Chemical. Initial work on the IR SIL microscope has been presented at the *3rd International Conference on Advanced Vibrational Spectroscopy* and at the *Eastern Analytical Symposium.*

Future Plans: A thorough evaluation of all aspects of the IR SIL microscope performance is continuing. Particular focus will be placed in the following areas: demonstration of absorbance contrast, evaluation of the significance of the sample/SIL separation on image properties, and evaluation of SIL imaging with a thermal IR source of the type utilized in typical commercial microscopes